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Influence of Deformation on Recrystallization of an Yttrium Oxide Dispersion-Strengthened Iron Alloy (PM2000)**

By *Carlos Capdevila Montes** and *Harry K. D. H. Bhadeshia*

Mechanically alloyed and compacted dispersion-strengthened iron-base alloys are materials which have been severely deformed, though uniformly deformed. However, these materials show a strange behavior, that the recrystallization temperature drops dramatically when the compacted sample is lightly cold deformed. Controlled experiments have been conducted on PM2000, which is an yttrium oxide dispersion-strengthened iron alloy. This involved the study of grain structure evolution in samples which were systematically bent and then heat-treated. A bend test introduces a strain gradient and compressive or tensile deformation about the neutral axis. It has therefore been possible to characterize the recrystallisation behavior in both tension and compression in a single test, with varying levels of strain. The results imply that any phenomenon which leads to a non-uniformity into the microstructure stimulates recrystallization with large effects on the evolution of the grain microstructure.

An alloy can be created without melting, by violently deforming mixtures of different powders.[1] Inert oxides can, using this technique, be introduced uniformly into the microstructure. The dispersion-strengthened alloyed powders are then consolidated using hot-isostatic pressing and extrusion, to produce a solid with a very fine grain structure. Heat treatment then induces recrystallization, either into a coarse columnar grain structure or into a fine, equiaxed set of grains. Columnar grains occur for two reasons: the oxide particles tend to become aligned along the extrusion direction, making that a favored growth direction. Alternatively, and in the absence of particle alignment, columnar growth can be stimulated by recrystallizing in a temperature gradient; the latter may be a stationary gradient or one which moves along the sample, as in zone annealing. The columnar microstructure is desirable in applications where the resistance to creep deformation is paramount.[2]

The ferritic oxide dispersion-strengthened alloy PM2000 is manufactured using the mechanical alloying process.[3–5] The development of a coarse grained microstructure during the recrystallization of PM2000, has been noted and discussed by a number of authors,[6–8] but the mechanism of grain control remains uncertain. Recent work on MA957, another mechanically alloyed ODS ferritic steel, has emphasized the large influence of non-uniformities on the development of the recrystallized microstructure.[9–11] Thus, it is argued that anything which introduces a heterogeneity into the microstructure stimulates recrystallization. This has the effect of greatly reducing the recrystallization temperatures and of enhancing the nucleation of recrystallization thereby giving an undesirable, fine-grained microstructure with poor creep properties. The heterogeneity can be introduced by having a non-uniform starting microstructure or by introducing a non-uniform plastic strain in the sample.

The purpose of the present work was to study the latter effect. PM2000 is sometimes used in tube form; the process by which the tubes are manufactured can introduce non-uniform plastic strains, which may radically alter the recrystallized microstructure. It is therefore important to understand the influence of strain heterogeneities in determining both the scale and anisotropy of the recrystallized microstructure.

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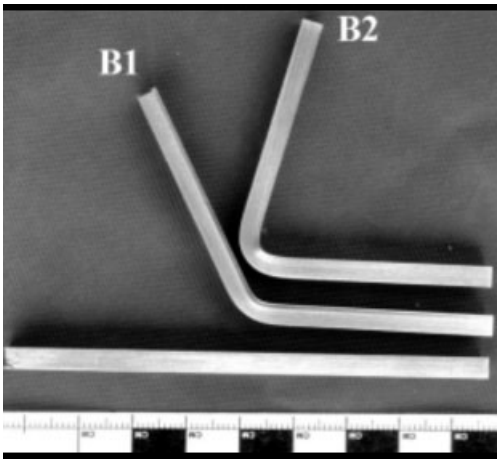

Fig. 1. Bend test samples with different levels of deformation.

The work presented here is inspired by Regle and Alamo,[11] who studied the recrystallization behavior of MA956 and MA957, for samples which were cold deformed after extrusion. Two deformation processes were used, swaging and drawing, with reductions ranging from 10–60 %. Swaging and drawing led to quite different changes in the recrystallization behavior. In all cases, deformation led to a reduction in the recrystallization temperature, the change being largest for the cold-drawn samples.

In the present work, PM2000 samples that were cold-deformed by bending after extrusion are studied. It is important to point out that material is already in a severly deformed state, although uniformaly deformed before further deformation during the bending test. Bend test samples are ideal for simulating deformation gradients. Table 2 and Figure 1 summarize the bend-test parameters for the two different levels of deformation implemented (B1 and B2).

The following analysis[12] deals with the problem of bending strains in a bar assuming plane-strain conditions, which probably is a good approximation for the central portion of the sample, where there are no large changes in width. The radial strains are given by

$$\varepsilon_r = \frac{R}{4}\left(1 - \frac{R^2 - (h/2)^2}{r^2}\right) \quad (1)$$

where R is the internal radius of curvature, h is the thickness of the sample, and r is an arbitrary radius which takes values ranging from R to $R+h$. The radial strains are plotted in Figure 2 using the input data listed in Table 2. For bending the strain passes through zero halfway through the thickness of the sample at the neutral axis, which is consistent with the plot presented in Figure 2. Likewise, the strain *gradient* in B2 sample is clearly much higher than that of B1.

Recrystallization in iron-base ODS alloys occurs at exceptionally high temperatures, of the order of 0.9 of the melting temperature (in PM2000 the melting temperature T_m is 1756 K). Recrystallization in such alloys nucleates by the bowing of grain boundaries. With the sub-micrometer grain size of mechanically alloyed metals, the grain junctions

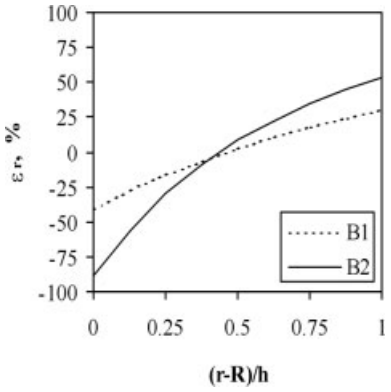

Fig. 2. Radial strains into the bend sample at room temperature.

themselves act as severe pinning lines for grain boundary bowing.[13] The activation energy for the nucleation of recrystallization is then very large so the few successful nuclei then give the very coarse-grained recrystallized microstructure. Deformation gradients assist nucleation by favoring some grains over others; the resulting increase in the nucleation rate leads to a finer recrystallized grain size together with a reduction in the recrystallization temperature, as shown in Figures 3 to 5. Whereas recrystallization does not occur after heat treatment at 1295 °C for 1 h in the unbent sample

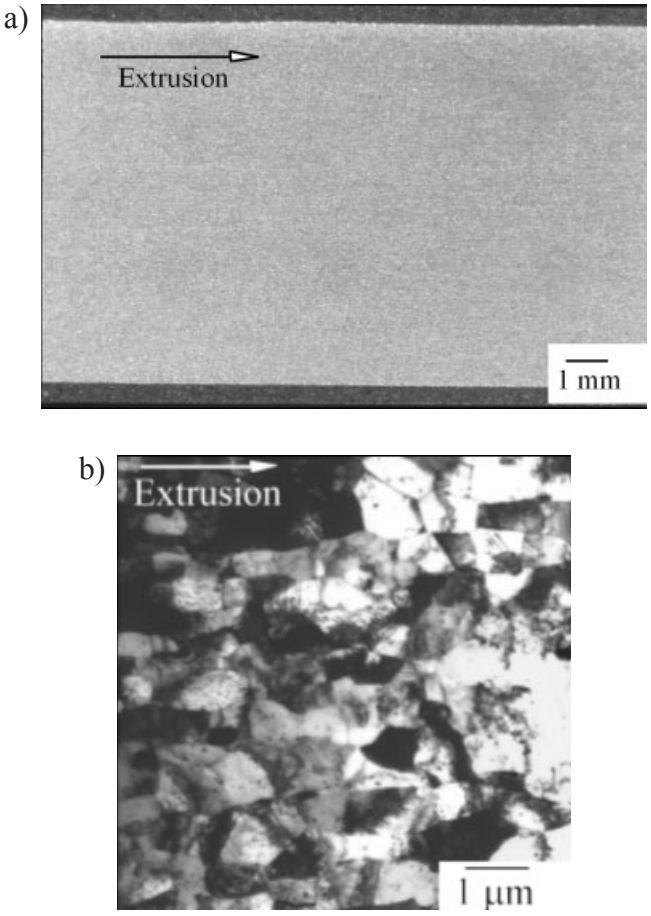

Fig. 3. a,b) Radial strains into the bend sample at room temperature.

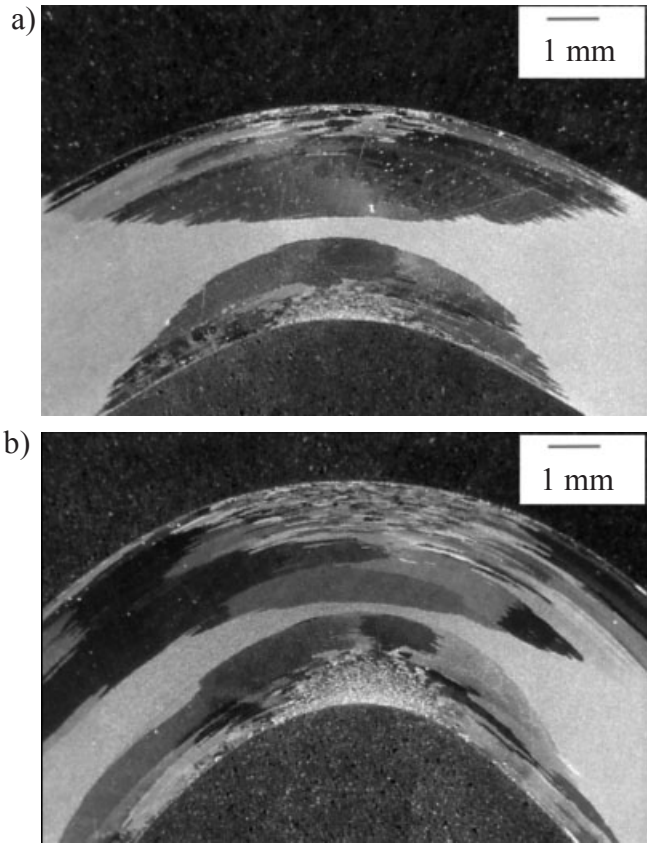

Fig. 4. Longitudinal section of unbent sample annealed at 1295 °C for 1 h. a) Optical image, and b) TEM image.

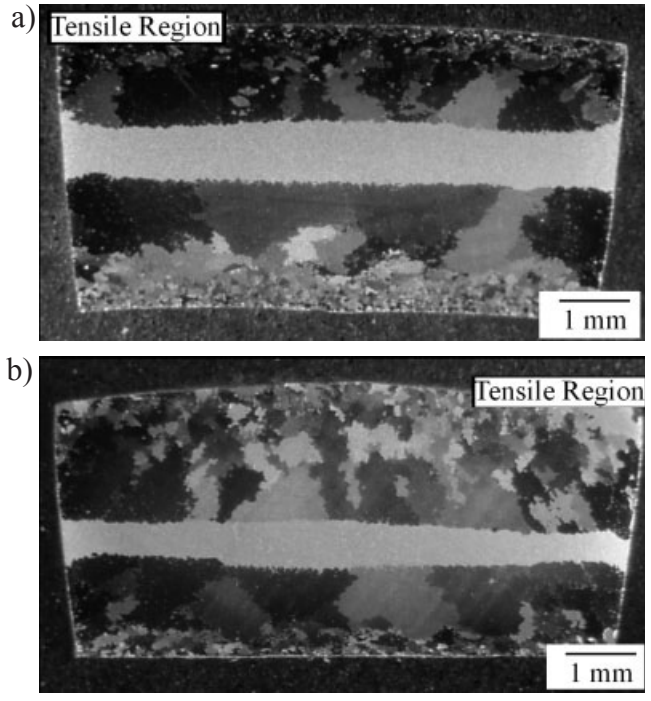

Fig. 5. Cross section of a) B1 and b) B2 bend samples recrystallized at 1295 °C for 1 h. The layer of light-etching material along the centreline is unrecrystallized.

(Fig. 3), B1 and B2 clearly show recrystallization in tensile and compression areas of the samples (Fig. 4).

Figure 5 shows the cross section of the B1 and B2 samples. It is clear that the center of the sample remains unrecrystallized since it corresponds to the neutral axis. Therefore, it is concluded from Figs. 3 to 5 that deformation clearly decreases the minimum temperature at which recrystallization begins. Moreover, it is clear from the comparison between Figures 4a and 5a, and Figures 4b and 5b the unrecrystallized area of the B2 sample is closer to the compression region than that of the B1 sample. This could indicate that the neutral axis approaches to the inner surface of the sample as bending becomes more severe. This is consistent with the bending theory where it is indicated that beyond the elastic limit the neutral axis moves closer to the inside surface of the bend as the bending proceeds.

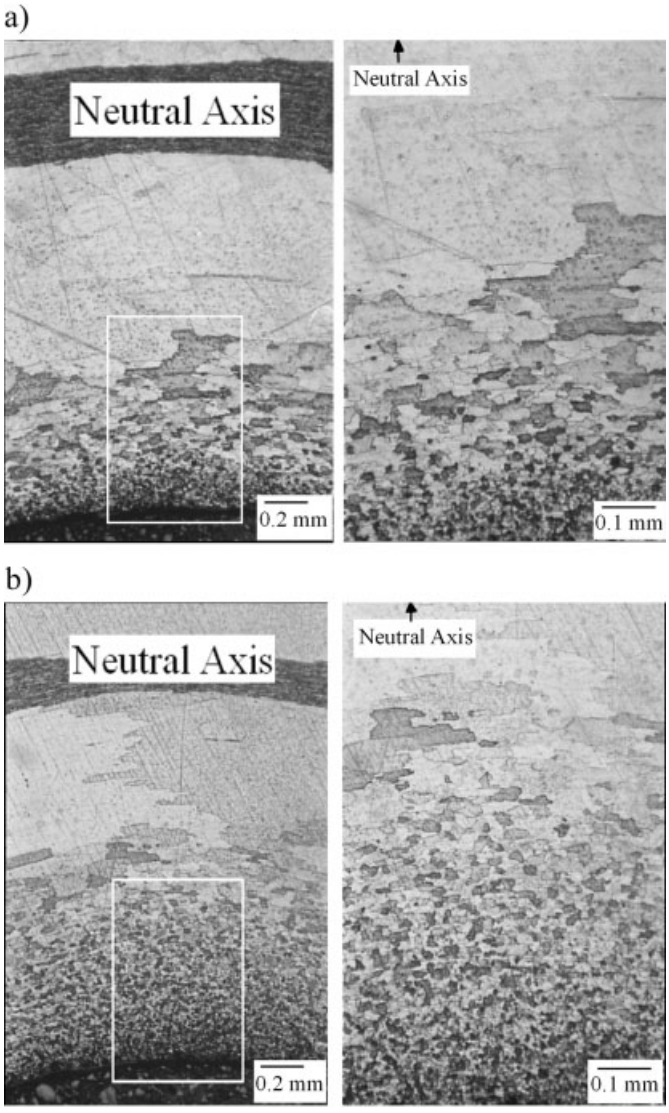

Fig. 6. Details of compression regions of a longitudinal section of (a) B1 and (b) B2 bend samples recrystallised at 1295 °C for 1 h. The area inside the square is enlarged on the right.

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The grain structures in the tensile and compression areas of the bend samples are presented in detail in Figures 6 and 7. The larger strain gradient of B2 leads to more a equiaxed and smaller recrystallized grain structure. The grains in the compressed inner regions (Fig. 6) are more equiaxed and refined than those in the tensile regions (Fig. 7), which is consistent with strain gradient analysis shown in Figure 2. Likewise, the recrystallized grains in the compression and the tension areas of B2 (Figs. 6b,7b) are finer than those in B1 (Figs. 6a,7a). These results are consistent with the idea that anything which introduces heterogeneity into the microstructure stimulates recrystallization.[9,10]

Since the grains in B2 sample becomes very fine and they can not be properly discriminated with optical image techniques, Figure 8 shows FEG-SEM image of the compressed inner, center and tensile outer parts of the B2 sample. The comparison between Figures 4 to 8 evidence the extreme variation on grain size due to the strain gradients introduced in the sample by bending. Likewise, Figure 8b shows the grain morphology in the neutral-axis region of the sample. It is clear that although no recrystallization has been produced, the grains are deformed resulting in an elongated structure when compared with the microstructure shown in Figure 3b.

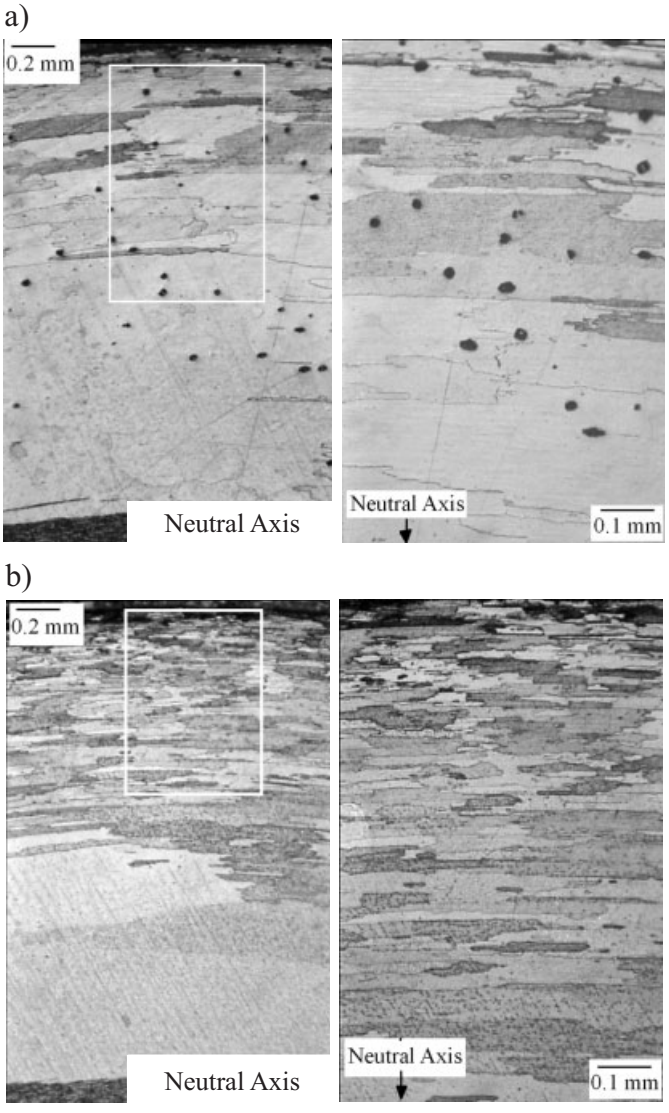

Fig. 7. Details of tensile regions of a longitudinal section of a) B1 and b) B2 bend samples recrystallised at 1295 °C for 1 h. The area inside the square is enlarged on the right.

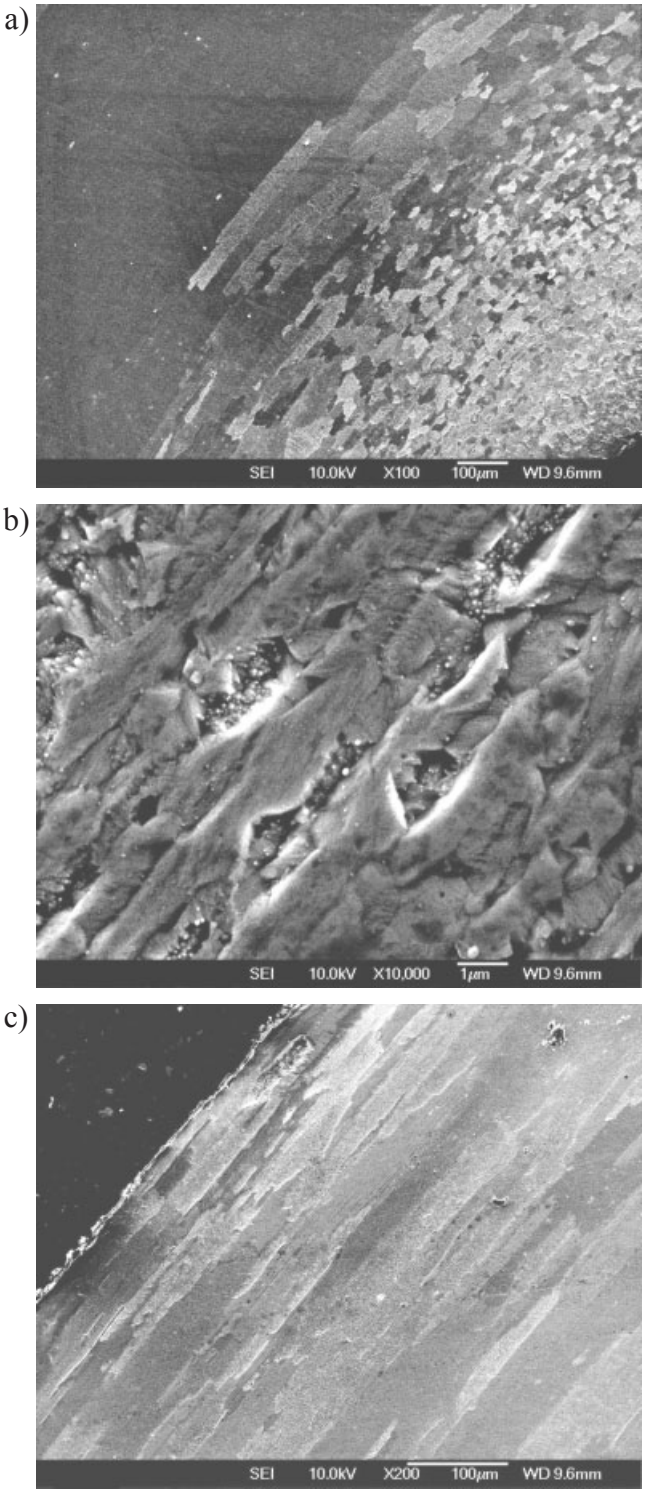

Fig. 8. Details of the secondary electron images (SEI) obtained from the a) compression, b) centre, and c) tensile regions of the B2 sample.

From these results it could be concluded that non-uniform deformation promotes the nucleation of recrystallization, and consequently leads to a finer grain microstructure in PM2000. However, PM2000 is designed for high temperature applications. High-temperature strength is enhanced by the development of a coarse-grain microstructure with a high aspect ratio. Therefore, more homogeneous deformation has to be produced in order to reduce nucleation, and then promote coarse grain structure.

The tensile properties of PM2000 have been analyzed using the neural network model created by Badmos et al.,[14] which has as inputs virtually all the parameters that are known to affect the strength. This includes the detailed chemical composition, any recrystallization or ageing heat-treatment, the extent of cold work, the test temperature, and the strain rate. Figure 9 shows the evolution of the yield strength with temperature for as-flow formed material. The solid line indicates the evolution of yield strength with temperature, meanwhile the upper and lower dashed lines indicate the range of uncertainty (error bars) of the predicted yield strength. There is a significant decrease in the yield strength at and beyond about 500 °C. Therefore, bending at 500 °C might induce more homogeneous deformation instead of that induced at room temperature where the yield strength is much higher. Figure 10 shows the microstructure obtained after recrystallization heat treatment at 1295 °C for one hour in the compressed inner and tensile outer regions of the B2 sample but bent at 500 °C instead of at room temperature. A notable difference in the grain structure as compare with that for room temperature is observed (Figs. 6b,7b). A more homogeneous and coarse grain structure is obtained.

The ferritic alloy PM2000 in the as-received condition had been subjected to severe but uniform deformation during manufacture. In this study, the effect of non-uniform deformation on the recrystallization behavior of PM2000 was shown to have a two fold effect. Firstly, the recrystallization temperature decreases, consistent with the hypothesis that anything that makes the original microstructure heterogeneous will encourage recrystallization. This is because the microstructure prior to recrystallization is relatively uniform with grains that are so fine their junctions are powerful pinning points. The second effect is that the increase in the number and density of recrystallization nuclei, due to (non-uniform) cold deformation, leads to fine grain structures, which are also more isotropic in three dimensions.

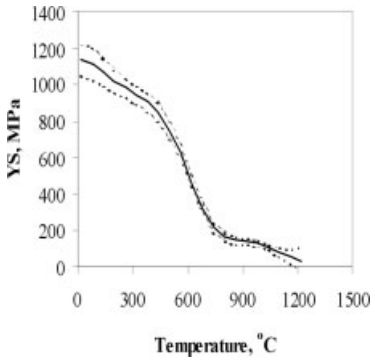

Fig. 9. Effect of the temperature on the yield strength (YS) of PM2000 calculated using the method of Badmos et al. [17].

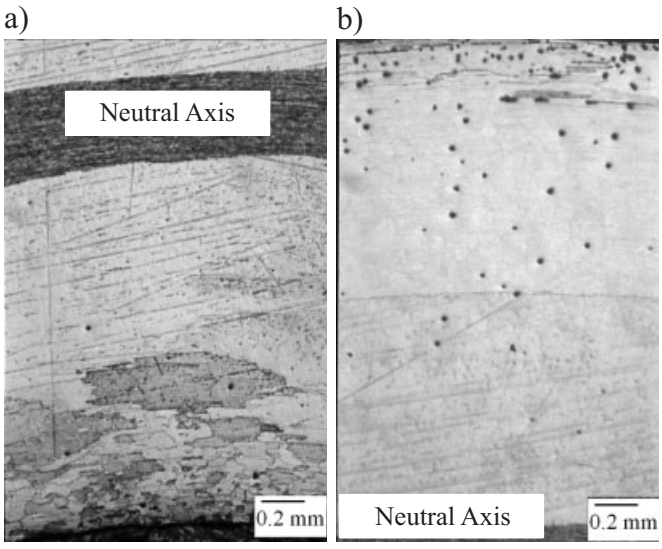

Fig. 10. Microstructure of the a) compression, and b) tensile region in a longitudinal section of a bend sample deformed at 500 °C.

If coarse, columnar grain structures are desirable in the context of creep strength, then the present work indicates that the processing of PM2000, or similar materials in order to produce tubes, should avoid plastic strain gradients. Indeed, it is predicted that coarse, directional grain structures are only expected when the deformation following consolidation is either zero or very large, since both cases lead to a uniform distribution of plastic strain.

Experimental

The nominal composition of the alloy PM2000 used in this work is shown in Table 1. The alloy was supplied by PLANSEE GmbH. The essential feature of PM2000 is that it contains 5.5 wt.-% of Al and 0.5 wt.-% of Y_2O_3. The aluminium enhances corrosion and oxidation resistance and it is claimed that PM2000 is better than other ODS in gaseous environments containing SO_2 0.24 %, CO_2 15 %, O_2 4 %, N_2 to balance [15,16]. The creep performance has been found to be optimum with a Y_2O_3 content of 0.5 wt.-% [17].

Bend tests were employed to evaluate the role of deformation on subsequent recrystallization of as-received PM2000 in the form of extruded tubes with 4.7 mm wall thickness. The bend samples were machined into bars, each of 100 mm length, parallel to the extrusion direction, and with a rectangular in cross-section of 7 mm width (w) and 4 mm thickness (h). Guided-bend tests were carried out using a MAYES-100 kN bend machine. The axis of the bend was oriented at 90° to the direction of extrusion. The specimen was placed over two rounded supports separated by a clearance (L) equal to 40 mm. The specimen was bent by applying a force through a deforming tool in contact with the specimen at the mid-length between supports.

An optical image was used to observe the microstructures after etching using a mixture of 2 g $CuCl_2$, 40 mL HCl, and 40–80 mL ethanol. Metallographic examination in longitudinal and cross sections taken along the center line of the specimens were carried out. A Jeol JSM6500 field emission gun scanning electron microscope (FEG-SEM) has been used to reveal the fine grain structures.

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Deep Drawing and Impact Extrusion of Magnesium Alloys at Room Temperature

By *Hans-Wilfried Wagener*,* *Jörg Hosse-Hartmann*, and *Reinhard Friz*

For lightweight design, magnesium alloys and magnesium metal matrix composites (MMCs) are expected to become important structural and engineering materials. Their specific strengths are halfway between aluminum and titanium. This is the case even when their relatively high price is taken into consideration Fig. 1).

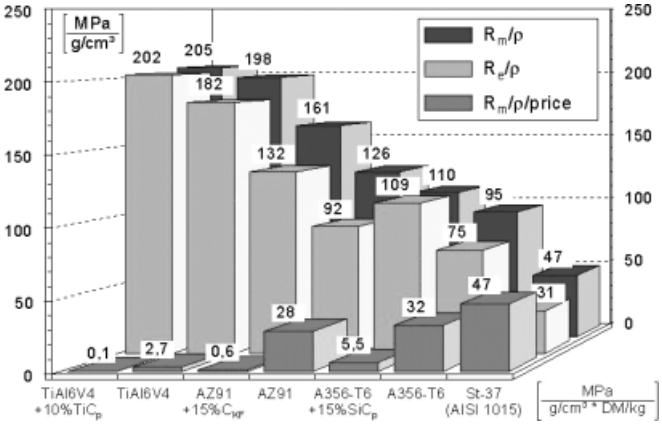

Fig. 1. Specific strength (strength/density) of light metals; R_m = ultimate tensile stress; R_e = yield stress; ϱ = density.

But a major problem with regard to the application of magnesium alloys in the automotive industry is their low ductility at room temperature. To avoid having to completely change the technologies of deep drawing and cold extrusion in the automotive industry, the principles of cold working of steels and of titanium and aluminum alloys must be applicable in the case of magnesium also. For this reason, a series of experiments are carried out to determine the forming behavior, the value of maximum strain, and the load-bearing capacity of the tested magnesium alloys.[1,2]

Deep Drawing: For the deep-drawing experiments, two types of hot-rolled AZ31 sheets of 1.5 mm thickness are tested. The wrought alloy AZ31 is selected, because it promises reasonable ductility at room temperature. In Table 1, the chemical composition of the AZ31 sheets are listed, and in Table 2 their mechanical properties are recorded.

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